

## Flotation of Potash for Carnallite Resources in Khur Playa of Iran Using Jameson Flotation Cell

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### Abstract

Carnallite processing using reverse flotation has been developed in recent decades. Khur Biabanak is the Iranian greatest Potash complex. Despite developments in the mineral processing technologies, common flotation systems which are used for potash flotation suffer from many disadvantages as high depreciation, wasteful energy usage and low production rate. In this project Jameson flotation cell is used and compared with current operating systems. In order to have an accurate comparison, a laboratory Denver cell is used in parallel. Under optimal circumstances for flotation of NaCl using Jameson cell recovery of 89.48% and grade of 83.24% are obtained. Concentration of Armoflote 619 as appropriate collector for NaCl flotation in optimum condition was 150 gr/ton and S/L ratio of 20% obtained as the best solid content. In the same situations in Denver cell 85.18% recovery for sodium Chloride with concentrate grade of 68.34% achieved as well.

**Keywords:** Carnallite; Flotation; Jameson flotation cell; Sodium chloride

### Introduction

Soluble salt minerals comprise a variety of industrially significant materials including alkali halides such as KCl and NaCl, alkali nitrates such as KNO<sub>3</sub> and NaNO<sub>3</sub>, alkali sulfates such as K<sub>2</sub>SO<sub>4</sub> and Na<sub>2</sub>SO<sub>4</sub>, and double salts such as carnallite (KCl.MgCl<sub>2</sub>.6H<sub>2</sub>O), kainite (KCl.MgSO<sub>4</sub>.3H<sub>2</sub>O), and schoenite (K<sub>2</sub>SO<sub>4</sub>.MgSO<sub>4</sub>.5H<sub>2</sub>O). Generally, these salts are used as potassium and nitrogen sources to produce agricultural fertilizers [1,2]. Potassium is one of the basic chemical ingredients used for promoting plant growth. Refined potash salts, obtained from either ores or brines, are the only economically significant sources of potassium used in fertilizers [3].

### Potash production

In addition to traditional potash ores, naturally occurring brines (e.g., the Dead Sea, the Great Salt Lake, etc.) contain dissolved potassium salts and are important secondary sources for commercial potash production. Nevertheless, the most important potash ore is sylvinite, a mixture of sylvite (KCl) and halite (NaCl) with minor amounts of clay and other potassium minerals.

Today, the majority of the sylvinite ores in the world are concentrated by froth flotation involving brines fully saturated with respect to potassium and sodium chlorides [1,4,5]. Shown in Table 1 are the world potash mine production and reserves (U.S. Geological Mineral Commodity Summaries, January 2018).

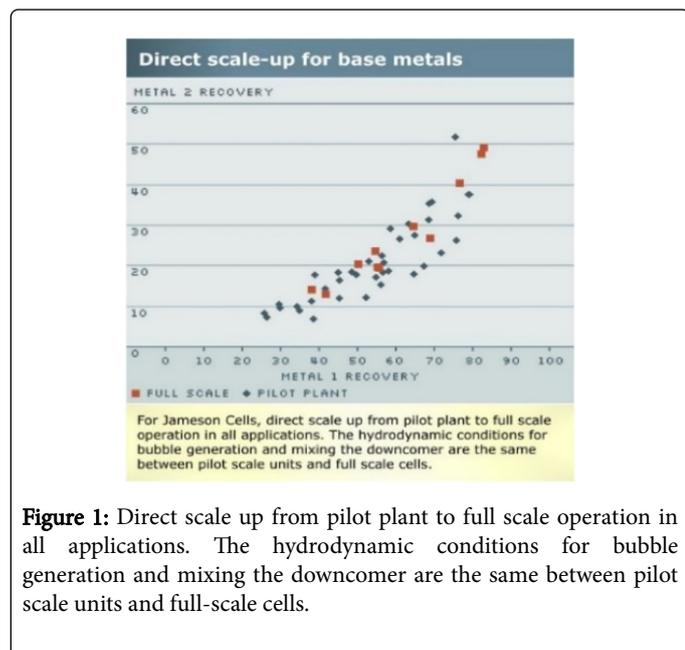
	Mine production		Reserves
	2016	2017	
United States	500	480	210,000
Belarus	6,180	6,400	750,000
Brazil	301	300	24,000
Canada	10,800	12,000	1,000,000
Chile	1,200	1,200	150,000
China	6,200	6,200	360,000
Germany	2,800	2,900	150,000
Israel	2,050	2,200	270,000
Jordan	1,200	1,300	270,000
Russia	6,480	7,200	500,000
Spain	670	680	44,000
United Kingdom	450	450	40,000
Other countries	480	500	90,000
World total (rounded)	39,300	42,000	3,900,000

**Table 1:** World potash mine production and reserves in thousand metric tons of K<sub>2</sub>O equivalent (U.S. Geological Mineral Commodity Summaries, January 2018).

### Jameson cell

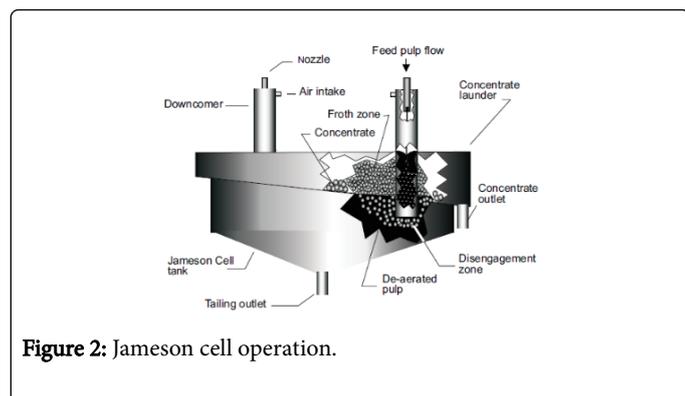
In recent years, many advances have been made in the field of mineral industry. New advancements all contribute to increasing recovery and reducing industrial costs. One of these advances in the

field of mineral processing is the construction of Jameson Flotation cells. These cells were first used by Professor Jameson in 1988 in coal flotation. Since 2000 these cells have rapidly replaced mechanical cells in different parts of the world. Jameson flotation cell having many advantages including high production capacity, fast and low-cost maintenance, low investment costs, direct scale up from pilot plant to full scale operation, high concentrate production, and high recovery is a serious rival for mechanical and column cells (Figure 1).



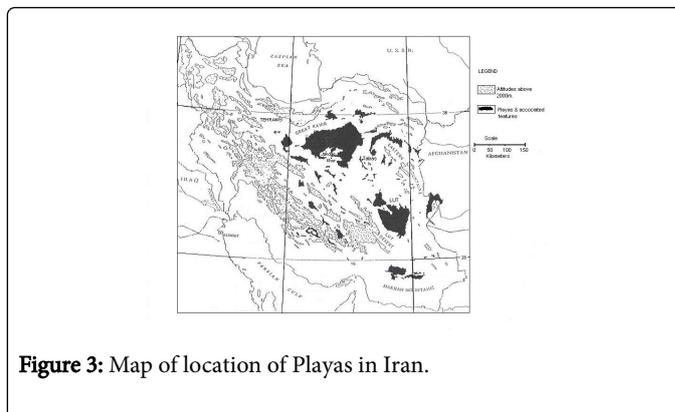
**Figure 1:** Direct scale up from pilot plant to full scale operation in all applications. The hydrodynamic conditions for bubble generation and mixing the downcomer are the same between pilot scale units and full-scale cells.

Furthermore, the Jameson cell is also used to separate the organic phase from aqueous phase in solvent extraction processes that indicates the importance and efficiency of this cell [4]. A Flowsheet of this cell is shown in Figure 2.



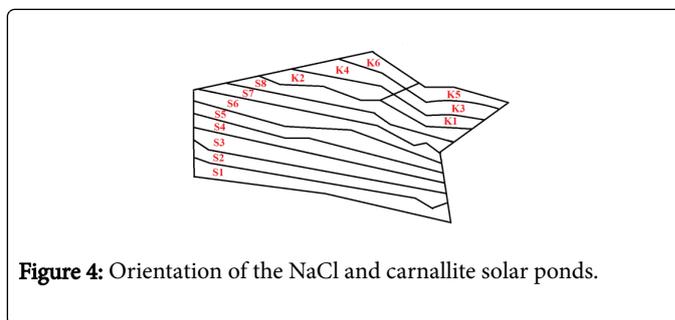
**Figure 2:** Jameson cell operation.

Brine mining and the characteristics of the playa of the Khur Biabanak: The Great Kavir is located in the center part of Iran. This region has low rainfall (86.5 mm/yr) and high evaporation (2857 mm/yr) property [6]. The most important and economical minerals such as halite and potash are deposited in the Great Kavir, especially in Khur Playa. This playa is located at the 310 kilometers of the east of Naïen and 40 kilometers of the east of Khur and in the north of Khur-Tabas road. Figure 3 shows the location of these playa in Iran.



**Figure 3:** Map of location of Playas in Iran.

Khur Playa with the extent of 2000 square kilometers has the thickness about 7.5 meters and the east-west slope. For this reason, at the time of rainfall, water moves from east to west. Because it is not surface water, to collect this water, a channel with the length of 45 km and the depth of 5 meters has been drilled at the western side of this area. The brine collecting channel sends the brine to the solar ponds, where crystallization of NaCl crystals occurs. The number of NaCl ponds with the letter S is 8 and the number of carnallite ponds with the letter K is 6.70% of the total area of the ponds is related to the precipitation of NaCl, and the remainder is related to the carnallite precipitation. The height of the pond walls is 2.5 meters and each pool have a height difference of 4 meters to the next pool. Orientation of the solar ponds is shown in the Figure 4.



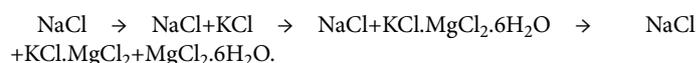
**Figure 4:** Orientation of the NaCl and carnallite solar ponds.

The density of NaCl precipitation is about 1.375 gr/cm<sup>3</sup>, then the condensed brine is sent to the carnallite crystallization ponds for carnallite crystallization. Carnallite would precipitate at the density of 1.440 gr/cm<sup>3</sup>. The carnallite is harvested by dredging. The chemical analysis for the carnallite mixed crystal sample is presented in Table 2.

NaCl	KCl	MgCl <sub>2</sub>	CaCl <sub>2</sub>
15.82	19.44	28.51	2.24

**Table 2:** Chemical analysis for a typical carnallite mixed crystal sample.

It can be seen from Table 2 that the brine from solar ponds contains K<sup>+</sup>, Na<sup>+</sup>, Mg<sub>2</sub><sup>+</sup> and Cl<sup>-</sup> which are crystallized in the following way:

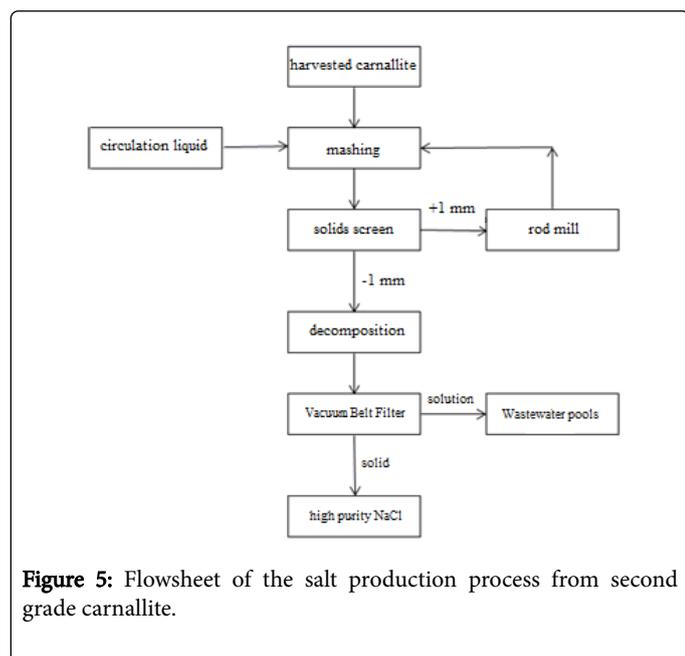


NaCl is always present with carnallite (KCl.MgCl<sub>2</sub>.6H<sub>2</sub>O) during the formation of carnallite and 8-20% NaCl has to be separated from carnallite in order to produce KCl from carnallite [7].

Currently there are two industrial ways to separate KCl from a mix of carnallite/NaCl crystals. The first way is flotation of KCl with an amine collector. In this method, before flotation the selective cold decomposition of carnallite would be done which keeps the KCl in the crystalline state and after cold decomposition flotation would separate KCl from NaCl. In the second way at first flotation would float NaCl from carnallite with an alkyl-morpholine collector and then KCl would be separated by cold decomposition of carnallite to produce KCl [8]. This paper is confined to the second strategy.

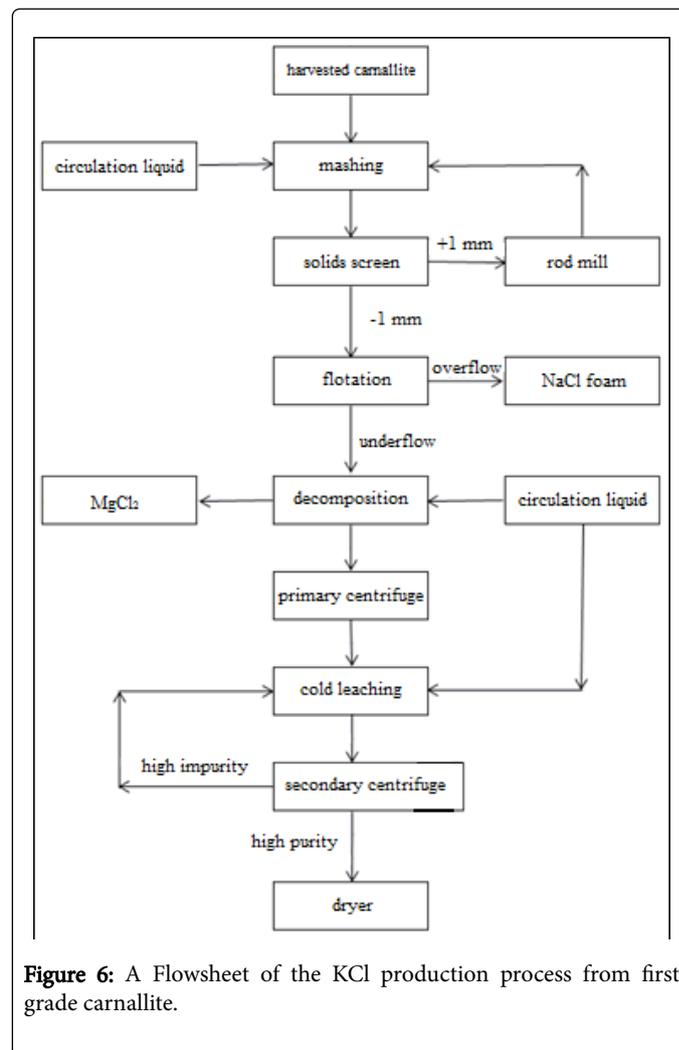
## Processing Steps

The inputs to the plant, according to the amount of potash contained therein, are classified into two categories of first and second grade carnallite. As can be seen in the Flowsheet of the factory process, to separate low-grade materials, with low KCl contents (type 2), the material is mixed with saturated brines in the reservoirs (mashing), and then, the pulp is sent to the screen (Figure 5). In this section, the coarse material is separated and routed to the rod mill for crushing and the finer materials are sent to the decomposition tank. A small amount of  $MgCl_2$  and KCl in the pulp, due to the large difference in the solubility of these two salts with sodium chloride, these two salts dissolve in water and the remaining materials are sent to the Vacuum Belt Filter, in this section the pulp is poured onto the cloth, while below it, the suction pumps will absorb the water in the pulp and NaCl, which is present in the pulp as insoluble, remains on the cloth.



For the separation of high-quality materials, (first grade carnallite), the operation has more details and steps. For this feed, after mixing the material at the mashing stage and passing the material out of sieve, the pulp is sent to a thickener in order to raise the solid content. After increasing the solid content, the pulp is sent to the conditioning tank; in this tank the collector is added to the pulp and inserted into the flotation cells after complete mixing. At this stage, NaCl is removed along with the froth from the pulp and the pulp containing  $MgCl_2$  and KCl is introduced into the decomposition tank from the underflow of the flotation section. In this tank, the water is added to the pulp, using

the difference in the solubility of  $MgCl_2$  and KCl,  $MgCl_2$  dissolves in water and the final pulp, which it's only remaining solid is potassium chloride, is directed toward centrifuges.



After separating solids in the centrifuge section, the solids are directed to the cold leaching tank. In this tank, if the purity of potassium chloride becomes desirable, the materials are continuously mixed and diluted and returned to the centrifuge section. After reaching the appropriate grade, the solids are separated and sent to the dryer to be completely dried and the final product will be obtained (Figure 6).

## Feed preparation

Feed required was selected from first grade carnallite. After grinding using a roller crusher, a sieve analysis was performed on samples. The results of the sieve analysis are presented in Table 3.

It should be noted that the input carnallite feed before the flotation stage must be thoroughly crushed and has dimensions below one millimeter. To reach these dimensions, the primary feed was passed through a series of 4 sieves and coarse particles were regrinded.

Sieve size (#)	Weight retained (gr)	Weight retained (%)	Cumulative % weight retained	Cumulative % weight passing
10	7	1.59	1.59	98.41
12	7	1.59	3.17	96.83
14	20	3.4	6.58	93.42
16	21	3.85	10.43	89.57
18	53	12.02	22.45	77.55
20	33	9.07	31.54	68.48
30	89	20.18	51.7	48.3
40	50	11.34	63.04	36.96
-40	163	36.96	-	-

Table 3: Feed sieve analysis.

### Solution preparation

One of the important steps before flotation is solution preparation. If the consumable solution is not saturated, then the feed into the cell will dissolve in water during flotation, and the particle size change will occur.

In order to prepare a circulating solution, first a certain proportion of water and carnallite must be mixed together, then, filter the solids in the solution to make the solution free from solids. The question that arises in this section is when the saturation of the solution can be ensured, because different salts have different degrees of solubility, it cannot be said that when the dissolution of carnallite in water has stopped, the solution has reached saturated state. Two methods have been proposed to achieve the appropriate state:

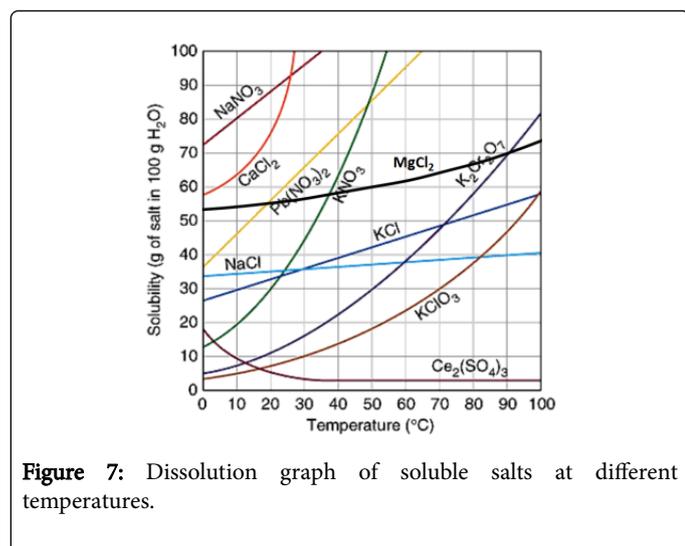


Figure 7: Dissolution graph of soluble salts at different temperatures.

**Density measurement:** This method is based on the experience gained at the factory. Accordingly, when the density of circulation solution reaches 1.284-1.284 kg/m<sup>3</sup>, the saturated solution is considered to be saturated. That means in spite of depositing the material and stopping the apparent dissolution, salt dissolution in

water should be continued until the density of the solution reaches to the limit.

**Dissolution of the material:** This method is proposed based on dissolution charts at different temperatures. Based on this chart, the amount of material that is dissolved in water at a given temperature is determined and considering the salt percentage in the feed, the dissolution of each salt in water can be determined. Figure 7 and Table 3 are presented for a better understanding of the topic.

Salt	Temperature					
	0°C	10°C	20°C	30°C	40°C	50°C
NaCl	35.65	35.72	35.89	36.09	36.37	36.69
KCl	28	31.2	34.2	37.2	40.1	42.6
MgCl <sub>2</sub>	52.9	53.6	54.6	55.8	57.5	59.2
CaCl <sub>2</sub>	59.5	64.7	74.5	100	128	132.5

Table 4: Salts dissolution table at various temperatures (gr/100 ml).

If the temperature be 20°C and the feed sample analysis be as follows (Table 4), the amount of material needed to prepare one liter of saturated solution can be calculated as follows:

Salt name	NaCl	KCl	MgCl <sub>2</sub>	CaCl <sub>2</sub>
%	28.9	21.5	28.9	1.45

Table 5: Flotation feed analysis.

The amount of sodium chloride dissolved in one liter of water:

$$100 \text{ gr feed}/X \text{ gr feed}=14.4 \text{ gr NaCl}/358.9 \text{ gr NaCl} \rightarrow X=2475.17 \text{ gr NaCl}$$

And in the same way: 1590.69 gr KCl, 1889.27 gr MgCl<sub>2</sub>, 51379.31 gr CaCl<sub>2</sub>.

Using the Table 5, we can determine the exact amount of salt dissolution in water. On the other hand, the analysis of the feed shows

that each of the salts is what percentage of the feed, for example, in the case of sodium chloride, it can be said that there is 14.4 gr NaCl per 100 gr of feed. Now according to the grades, it can be determined that what weight of feed contains 358 gr of NaCl. The highest amount indicates the amount of material needed to make the saturated solution, because if the substance that has the highest dissolution reaches the saturation level, of course, the other materials will also be saturated. It should be noted that, despite the fact that CaCl<sub>2</sub> has the highest content, due to the fact that its unsaturation does not cause any problems in flotation, it is not considered as the limiting material.

### Mashing

The purpose of this stage of the process is to remove CaCl<sub>2</sub> from the solid phase, because high concentrations of CaCl<sub>2</sub> disrupts the froth during the flotation stage. Table 6 shows the effect of CaCl<sub>2</sub> on flotation [8].

Concentration of CaCl <sub>2</sub> in solution (gr/lit)	NaCl Recovery (%)
30	87.22
75	88.09
135	76.83
180	62.44

Table 6: Effect of Calcium Chloride Concentration on Flotation.

At this stage, the circulation solution and the flotation feed are mixed in the conditioning tank so that the CaCl<sub>2</sub> present in the feed, which is much more soluble than the other salts, and the percentage of it is very low in the feed, would dissolve in solution. Of course, according to Table 6, the presence of CaCl<sub>2</sub> in the circulation solution is also harmful in high amounts, for this purpose, the percentage of circulating solution is replaced with fresh solution at each use of the circulating solution in the flotation to prevent excessive concentration of calcium chloride in the circulation solution.

### Collector preparation

The collector used in the tests is an alkyl-morpholine compound known as the Armoflote 619 brand. The morpholine is used as the flotation collector it is usually an alkyl-morpholine with a 12 – 22 carbon chain. A Flowsheet drawing of an alkyl-morpholine molecule is shown in Figure 8. The chemical properties of morpholine are similar to tertiary amines. These features are shown in Table 7.

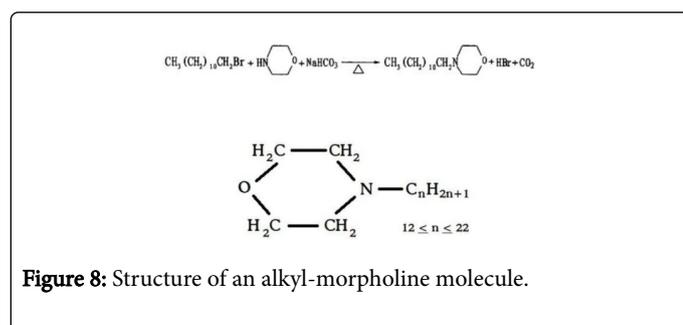


Figure 8: Structure of an alkyl-morpholine molecule.

Formula	C <sub>4</sub> H <sub>9</sub> NO-C <sub>n</sub> H <sub>2n+1</sub>
Molar mass	87 gr/mol
Appearance	Transparent liquid
Density	870 Kg/m <sup>3</sup>
Flash point	100
Boiling point	130-170
Solubility in water	Slightly soluble in water

Table 7: Properties of alkyl-morpholine.

With the synthesis of the morpholine and alkyl bromodecane, Alkyl-morpholines are obtained. But this process has some disadvantages like producing large amounts of CO<sub>2</sub> and the high synthesis time (about 12 h). The efficiency of this operation is about 84%. In 2001, a new method developed by Yu et al. [9]. Using this method cause a significant recovery in the efficiency of dodecyl morpholine production. In this method the reaction divides into two stages. In the first stage dodecyl-bromodecane and potassium hydroxide are mixed at the heat of 150°C and then morpholine will be added to produce dodecyl morpholine. Using this method, the time of synthesis is reduced to 4-6 h and the efficiency is improved to 95%.

In order to prepare this collector, a certain amount of circulating solution and the collector should be heated up to 50°C, and then the pH of the solution should adjust to 5 by adding hydrochloric acid.

### Flotation

The inverse flotation, with the use of alkyl morpholine, has special conditions. In these experiments, NaCl floats and comes up with the froth. On the other hand, researches show that this collector is able to float both NaCl and KCl. If the carnallite in the pulp decomposes during flotation, as shown in Figure 9, then carnallite decomposes into MgCl<sub>2</sub> and KCl, will float along with NaCl. For this purpose, it is important to control the saturation of the solution prior to the flotation, in order to ensure that the carnallite is not decomposed in the flotation stage [10].

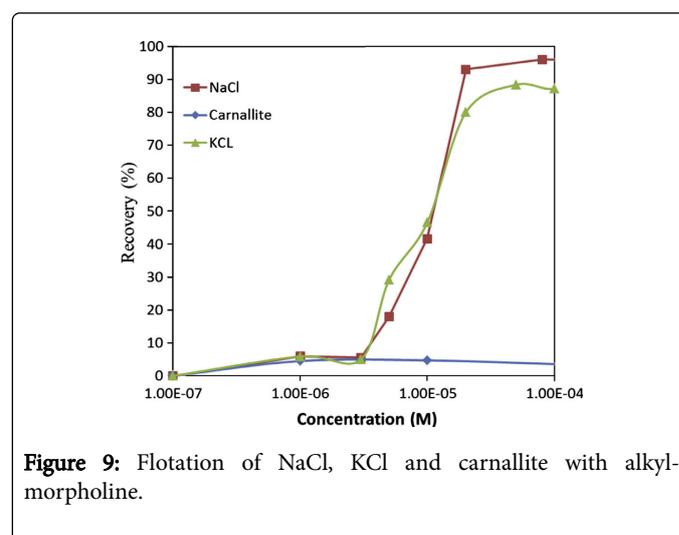


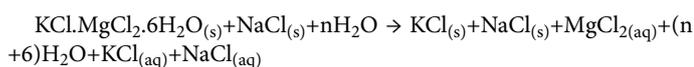
Figure 9: Flotation of NaCl, KCl and carnallite with alkyl-morpholine.

Flotation tests were done in mechanical and Jameson cell. For Denver cells three parameters of collector concentration, solid percent, and the ratio of fresh solution to circulating solution were considered as variable parameters. Based on this, Denver cell experiments were designed using the DX7 software at two levels up and down, for the collector concentration parameter between 50 and 250 grams per ton, for the solids percentage of between 15 and 25 percent, and for a ratio of fresh solution to circulating solution of between 0 and 80 percent. Experiments were done in a mechanical flotation cell with a volume of 1500 ml. In all experiments, the mixing speed was 1500 rpm and the time of every test was 10 minutes. After preparing the required solution, the pulp was mixed for 4 minutes in order to prepare and perform the mashing stage. The collector was then added to the pulp and 3 minutes was taken for the complete mixing of the chemicals in the pulp [11-15]. Then aeration started and the resulting foam was collected for 3 minutes. Then the overflow and underflow were separated from the solution using a filter press. The samples were then dried at 60°C using a drying machine and dry weight was recorded. Flame Photometer was used to analyze the samples.

For Jameson cell, tank diameter and the downcomer depth in the tank were selected as two hydrodynamic variable parameters. The downcomer has a height of about 3 meters and a diameter of 5 cm. the flowrate of pulp is about 30 liters per minute. Aeration rate was considered to be 6 liters per minute. With this condition, the air-to-pulp ratio will be 0.2, which is optimal for the Jameson Flotation experiments. The solid content of each experiment was 15% and the time of each test was 10 minutes. Preparation of experiments were done in a tank with the volume of 120 liter and 70-liter circulation solution were used in every test.

### Cold decomposition

After flotation cold decomposition is done for purifying the final product. Cold decomposition controls and upgrades the overall recovery and product quality. In this stage, at ambient temperature (temperature of 10-25°C), carnallite decomposes to KCl and MgCl<sub>2</sub> by the following reaction:



NaCl in the written reaction is the remained NaCl that the flotation system could not separate it. In this stage, in order to achieve to the final product, carnallite must be broken to its components with adding water. If the added water to the system is not sufficient the carnallite cannot be completely decomposed. If the water addition is too much, then some KCl will be dissolved during the decomposition stage. Therefore, water addition is so important in this stage. Usually water equivalent to 40% of the carnallite weight is used [16-20]. After decomposition of carnallite, because the solubility of MgCl<sub>2</sub> is more than KCl, as shown in Figure 7, most of the existing MgCl<sub>2</sub> is dissolved in water and the only remaining solids will be KCl. Then this pulp is sent to the primary centrifuges to separate the solid phase from the liquid phase. At this stage the grade of produced KCl is upper than 90%. The remaining solids will be sent to the cold leaching tank. In this tank, for the complete separation of impurities, water is added again to the pulp and the resulting pulp is sent to the secondary centrifuges to produce a final product with a purity of 99.5%.

## Results

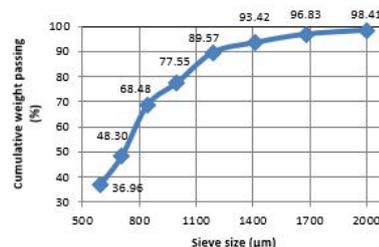


Figure 10: Chart of feed sieve analysis.

Figure 10 shows the results of the sieve analysis of the feed. According to the above diagram, the d80 value is approximately 1000 microns and the d50 value is about 700 microns.

### Denver cell

Results of the Denver flotation tests are shown in Figures 11 and 12.

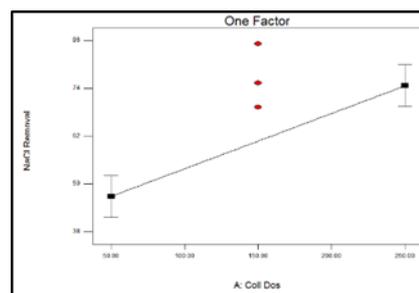


Figure 11: Effect of collector concentration on NaCl recovery.

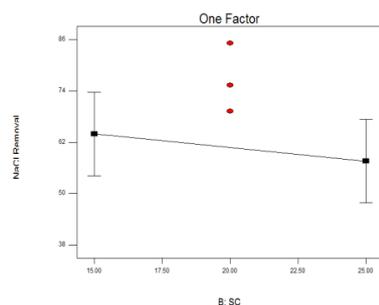


Figure 12: Effect of solid content on the recovery of NaCl.

It can be seen that increasing the collector concentration has increased salt recovery, but the best result is seen at a concentration of 150 gr/ton collector and a solid content of 20%. The reason for this may be due to the excessive increase in the amount of collector that can cause hemi micelle phenomenon, because there are also quantities of collectors in the circulation solution, and this may have negative effect when the concentration of collector is 250 gr/ton in the test and cause a reduction on NaCl recovery [21-25].

Another reason may be due to the excessive percentage of solid content in a cell with the volume of 1500 ml. It can be said that the cell may not have the ability to perform tests with high solid contents. In the other words when the solid content of a pulp becomes more than the cell's capacity, the turbulence that occurs in the cell causes loss of calm and prevents the material from rising and also detachment of the particles that are absorbed by collector.

### Jameson cell

In order to compare these two methods, the conditions in which the best results in Denver Flotation tests were obtained, were considered as constant parameters in the Jameson Flotation Tests. So, all tests are done with the solid content of 20% and concentration of 150 gr/ton collector and the test duration of 10 minutes.

As mentioned, two hydrodynamic variable parameters of tank diameter and the downcomer depth in the tank were selected to be studied [26-29]. Table 8 shows the results of the tests done in these conditions.

Test number	Tank (cm)	diameter	Downcomer dip in the (cm)	NaCl recovery (%)
1	15	40	33.56	
2	15	25	57.89	
3	25	32	67.65	
4	25	25	89.48	

Table 8: The results of the Jameson Flotation Experiments.

The results show that increasing the diameter of downcomer had a positive effect on NaCl recovery; the reason for this can be stated that as the diameter increases, more relaxation occurs inside the tank and the recovery of the material increases. Since there is a need to carry the material by the collector to achieve further recovery, so changing the diameter of the tank will causes a better rise of the materials in the tank. The second parameter is the depth of the downcomer in the tank. It can be seen that with increasing the distance between the downcomer and the end of the tank, the recovery increases. The reason may due to the decrease of the turbulence at the end of tank. It has also been proven that the spin of particles in the cone of the tank is much larger than the rest, so stopping too much of the material in this part will cause detachment of the particles from the collector and reduce the recovery. Also, by raising the downcomer, the retention time and the velocity of rotation of the particles along the bubble inside the tank will decrease, and the less time is spent in the tank, the more recovery will be achieved [30,31].

By comparing the performance of two cells, it can be said that using the Jameson Flotation cell has increased recovery by 4.3%. Also, the grade of the concentrate has been increased by 14.9%. Due to the production of fine bubbles, and high intensity and collision, and the high probability of bubble and particle contact, both recovery and grade are increased. Researches show that the bubbles produced in the Jameson Flotation cell have dimensions ranging from 0.3 to 0.5 mm, while the dimensions in the Denver cell are about 1 mm [32,33]. As the bubble rate increases per unit area, the possibility of the particles attachment with bubbles would be increased, and thus recovery will increase (Figure 13).

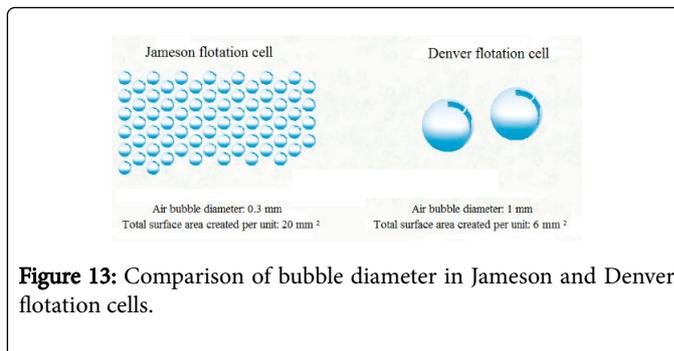


Figure 13: Comparison of bubble diameter in Jameson and Denver flotation cells.

Also, in the same time of flotation the Jameson cell used 22.47 kg feed while the Denver cell used 0.4 kg feed; which indicates the high capacity and production rate of the Jameson flotation cell.

### Conclusions

The aim of this project is to compare Jameson cell with the conventional Denver flotation systems. For this purpose, several Denver flotation tests were done to achieve the best conditions and examine the max ability of this system to recover NaCl foam from this special kind of brine. Results from these tests shows that the max recovery was achieved in the solid content of 25% and 150 gr/ton Armoflote 619 collector dosage. Then these conditions were applied on the Jameson flotation system to compare the grade and recovery of these systems. The analyzes of the final foams shows that the Jameson cell's recovery was 89.48% with the grade of 83.24% while Denver cell could recover 85.18% sodium Chloride with concentrate grade of 68.34%.

Due to the other benefits of the Jameson flotation cell, such as less space occupancy than other cells, require less maintenance due to lack of moving parts such as stirrers and rotors, less energy consumption, simplicity in converting a laboratory sample into Industrial sample due to uniform laboratory and industrial conditions, replacing this cell with the current flotation cells is highly recommended.

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